Federal Democratic Republic of Ethiopia

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ET ISO 3706 (1976) (English):
Phosphoric acid for industrial use (including foodstuffs) -- Determination of total phosphorus (V) oxide content -- Quinoline phosphomolybdate gravimetric method

ISO INSIDE

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ETHIOPIAN STANDARD

ES ISO 3706:2012

First edition

Phosphoric acid for industrial use (including foodstuffs)— Determination of total Phosphorus (v)oxide content - Quinoline phosphomolybdate grametric method

ICS: 67.220.20

Published by Ethiopian Standards Agency

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Foreword

This Ethiopian Standard has been prepared under the direction of the Technical Committee for

Food additives and contaminants (TC 21) and published by the Ethiopian Standards Agency (ESA).

The standard is identical with ISO 3706:1976 “Phosphoric acid for industrial use (including foodstuffs)- Determination of total Phosphorus (v) oxide content - Quinoline phosphomolybdate grametric method”, published by the International Organization for Standardization (ISO).

For the purpose of this Ethiopian Standard, the adopted text shall be modified as follows.

- The phrase “International Standard” shall be read as “Ethiopian Standard”; and
- A full stop (.) shall substitute comma (,) as decimal marker.

Phosphoric acid for industrial use (including foodstuffs) — Determination of total phosphorus(V) oxide content — Quinoline phosphomolybdate gravimetric method

1 SCOPE

This International Standard specifies a gravimetric method using quinoline phosphomolybdate for the determination of the total phosphorus(V) oxide content of phosphoric acid for industrial use (including foodstuffs).

2 FIELD OF APPLICATION

The method is applicable to phosphoric acids, whether or not they are homogeneous and whether or not they contain polyphosphoric acids.

3 PRINCIPLE

Preliminary hydrolysis of the polyphosphoric acids by boiling in the presence of hydrochloric acid. Precipitation of the phosphoric acid in the form of quinoline phosphomolybdate in the presence of acetone. Filtration, washing, drying and weighing of the precipitate.

4 REAGENTS

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % (*m/m*) or approximately 12 N solution.

4.2 Citromolybdate reagent.

4.2.1 Dissolve 70 g of sodium molybdate dihydrate ($Na_2MoO_4.2H_2O$) in 150 ml of water.

4.2.2 Dissolve 60 g of citric acid monohydrate ($C_6H_8O_7.H_2O$) in 150 ml of water and add 85 ml of nitric acid solution, ρ approximately 1,40 g/ml, about 68 % (*m/m*) or approximately 14 N solution.

4.2.3 Add, while stirring, solution 4.2.1 to solution 4.2.2.

4.2.4 Add 35 ml of nitric acid solution, ρ approximately 1,40 g/ml, about 68 % (*m/m*) or approximately 14 N solution, then 5 ml of recently distilled quinoline to 100 ml of water.

4.2.5 Add solution (4.2.4) to solution (4.2.3) and mix.

Allow to stand for at least 12 h and filter through the filter crucible (5.1).

Store this solution protected from light, in a well-stoppered flask.

4.2.6 Add 280 ml of acetone to solution (4.2.5) and dilute to 1 000 ml with water.

Do not keep this solution for more than 1 week. Store under the same conditions as solution (4.2.5).

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Filter crucible, with sintered glass disk, of porosity P10 (pore size index between 4 and 10 μm).

5.2 Electric oven, capable of being controlled at 250 ± 10 °C.

6 PROCEDURE

6.1 Test portion and preparation of the test solution

6.1.1 *Homogeneous phosphoric acid* (or phosphoric acid containing a precipitate which readily forms a suspension)

Weigh by difference, to the nearest 0,000 2 g, 5 ± 0,2 g of the test sample, in such a way that there is no gain or loss of moisture.

Transfer the test portion to a flask of about 250 ml capacity, add 10 ml of the hydrochloric acid solution (4.1), cover with a clock-glass and boil for about 10 min. Cool, add about 100 ml of water and 10 ml of the hydrochloric acid solution (4.1). Transfer the solution quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

Transfer 50,0 ml of this solution to a 500 ml one-mark volumetric flask, dilute to the mark and mix (solution A).

Prepare this dilution at the time of use.

6.1.2 *Non-homogeneous phosphoric acid,* containing a precipitate which does not readily form a suspension

Weigh by difference, to the nearest 0,05 %, the whole of the assay sample[1], in such a way that there is no gain or loss of moisture.

Transfer the sample to a polyethylene or polypropylene flask of suitable capacity, and homogenize by dilution or any other method which does not involve any loss of the constituents.

After homogenizing, weigh by difference, to the nearest 0,05 %, a test portion containing about 5 g of the original phosphoric acid, place in a 250 ml beaker and add 10 ml of the hydrochloric acid solution (4.1) cover with a clock-glass and boil for about 10 min. Cool, add about 100 ml of water and 10 ml of the hydrochloric acid solution (4.1). Transfer the solution quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

Transfer 50,0 ml of this solution to a 500 ml one-mark volumetric flask, dilute to the mark and mix (solution A).

Prepare this dilution at the time of use and filter if necessary.

NOTE — Homogenization may generally be achieved by simple dilution with water (or dilute hydrochloric acid solution), with prolonged stirring in a stoppered flask, the quantity of liquid added being weighed to the nearest 0,05 %.

When this treatment is insufficient, a more effective treatment should be carried out depending on the particular case (treatment with hot water with stirring in a stoppered flask, for example).

Finally, in the case where an insoluble deposit remains which adheres to the walls of the vessel containing the assay sample or which cannot be dispersed, it should be recovered, weighed and analysed separately.

6.2 Blank test

Carry out a blank test at the same time as the determination, following the same procedure and using the same quantities of all the reagents as used in the determination.

6.3 Determination

Depending on the expected phosphorus(V) oxide content, transfer to a 400 ml beaker the aliquot portion of solution A (6.1) shown in the following table.

Expected P_2O_5 content	Aliquot part of solution A (6.1) to be taken
% (*m/m*)	ml
Less than 30	50
From 30 to 50	25
Greater than 50	20

Dilute to about 100 ml and add 100 ml of the citro-molybdate reagent (4.2). Cover the beaker with a clock-glass and warm, inside a fume cupboard, on a hot-plate, until its contents reach 75 ± 5 °C and maintain at this temperature for about 30 s. (Do not use a flame and do not mix, either during the addition of the reagent or during the heating, so as to avoid the formation of clots.) Allow to cool to ambient temperature, stirring three or four times with a glass rod during cooling.

Heat the filter crucible (5.1) in the oven (5.2) controlled at 250 ± 10 °C, and leave for 15 min starting from the stabilization of the temperature. Allow to cool in a desiccator containing silica gel in good condition and weigh to the nearest 0,000 1 g.

Decant the liquid through the filter crucible (5.1) and wash the precipitate six times, by decantation, using about 30 ml of water each time. Transfer the precipitate quantitatively to the filter crucible (5.1) with the aid of a jet of water from a wash-bottle. Then wash the precipitate four times, removing each portion of wash water by suction.

Place the filter crucible in the oven (5.2) maintained at 250 ± 10 °C and leave for 15 min starting from the stabilization of the temperature. Allow to cool for not more than 30 min in a desiccator containing silica gel in good condition and weigh to the nearest 0,000 1 g.

7 EXPRESSION OF RESULTS

7.1 Method of calculation and formulae

The total phosphorus(V) oxide content, expressed as a percentage by mass of P_2O_5, is given by the formulae :

7.1.1 *Homogeneous phosphoric acid* (see 6.1.1)

$$(m_1 - m_2) \times 0{,}032\,07 \times \frac{500}{50} \times D \times \frac{100}{m_0} = $$

$$= \frac{32{,}07 \times D \times (m_1 - m_2)}{m_0}$$

7.1.2 *Non-homogeneous phosphoric acid* (see 6.1.2)

$$(m_1 - m_2) \times 0{,}032\,07 \times \frac{500}{50} \times D \times \frac{100}{m_0 \times \dfrac{m_4}{m_0 + m_3}} =$$

$$= \frac{32{,}07 \times D \times (m_1 - m_2) \times (m_0 + m_3)}{m_0 \times m_4}$$

where

m_0 is the mass, in grams, of the test portion (6.1);

m_1 is the mass, in grams, of precipitate obtained with the aliquot portion of the test solution (6.3);

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[1] The definition of the term "assay sample" will be given in ISO 4285, *Phosphoric acid for industrial use — Sampling technique. (At present* at the stage of draft.)

If the assay sample is not available, a test sample may be taken from the laboratory sample, which must first be thoroughly mixed so as to render it completely homogeneous. In this case, weigh, to the nearest 0,05 %, about 25 g and treat it as described for the assay sample.

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m_2 is the mass, in grams, of precipitate obtained with the corresponding aliquot portion of the blank test solution (6.2);

m_3 is the total mass, in grams, of water or other reagents added for the homogenization (see 6.1.2, note);

m_4 is the mass, in grams, of homogenized assay sample taken (see 6.1.2);

D is the ratio of the volume of the test solution (solution A) (6.1) to the volume of the aliquot portion taken for the determination (6.3);

0,032 07 is the factor for conversion of quinoline phosphomolybdate to phosphorus(V) oxide.

8 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard or regarded as optional.

ANNEX

PUBLICATIONS RELATING TO PHOSPHORIC ACID FOR INDUSTRIAL USE

ISO 847 – Determination of sulphate content – Titrimetric method.

ISO 848 – Determination of calcium content – Titrimetric method.

ISO 849 – Determination of iron content – 2,2′-Bipyridyl photometric method.

ISO 2997 – Determination of sulphate content – Method by reduction and titrimetry.

ISO 3359 – Determination of arsenic content – Silver diethyldithiocarbamate photometric method.

ISO 3360 – Determination of fluorine content – Alizarin complexone and lanthanum nitrate photometric method.*

ISO 3361 – Determination of soluble silica – Reduced molybdosilicate photometric method.

ISO 3706 – Determination of total phosphorus(V) oxide content – Quinoline phosphomolybdate gravimetric method.*

ISO 3707 – Determination of calcium content – Flame atomic absorption method.*

ISO 3708 – Determination of chloride content – Potentiometric method.*

ISO 3709 – Determination of nitrogen oxides content – 3,4-Xylenol photometric method.*

ISO 4285 – Sampling technique.

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* Also applicable to phosphoric acid for use in the foodstuffs industry.

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Ethiopian Standards Agency

Organization and Objectives

The Ethiopian Standards Agency (ESA) is the national standards body of Ethiopia established in 2010 based on regulation No. 193/2010.ESA is established due to the restructuring of Quality and Standards Authority of Ethiopia (QSAE) which was established in 1970.

ESA's objectives are:-

- Develop Ethiopian standards and establish a system that enable to check weather goods and services are in compliance with the required standards,
- Facilitate the country's technology transfer through the use of standards,
- Develop national standards for local products and services so as to make them competitive in the international market.

Ethiopian Standards

The Ethiopian Standards are developed by national technical committees which are composed of different stakeholders consisting of educational Institutions, research institutes, government organizations, certification, inspection, and testing organizations, regulatory bodies, consumer association etc. The requirements and/ or recommendations contained in Ethiopian Standards are consensus based that reflects the interest of the TC representatives and also of comments received from the public and other sources. Ethiopian Standards are approved by the National Standardization Council and are kept under continuous review after publication and updated regularly to take account of latest scientific and technological changes.
Orders for all Ethiopian Standards, International Standard and ASTM standards, including electronic versions, should be addressed to the Documentation and Publication Team at the Head office and Branch (Liaisons) offices. A catalogue of Ethiopian Standards is also available freely and can be accessed in from our website.

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International Involvement
ESA, representing Ethiopia, is a member of the International Organization for Standardization (ISO), and Codex Alimentarius Commission (CODEX). It also maintains close working relations with the international Electro-technical Commission (IEC) and American Society for Testing and Materials (ASTM).It is a founding member of the African Regional Organization for standardization (ARSO).

More Information?
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